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Title:	Micro-X-ray Fluorescence (MXRF) Direct Solids
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Los Alamos
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EST. 1943

Micro-X-ray Fluorescence (MXRF) Direct Solids

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Outline

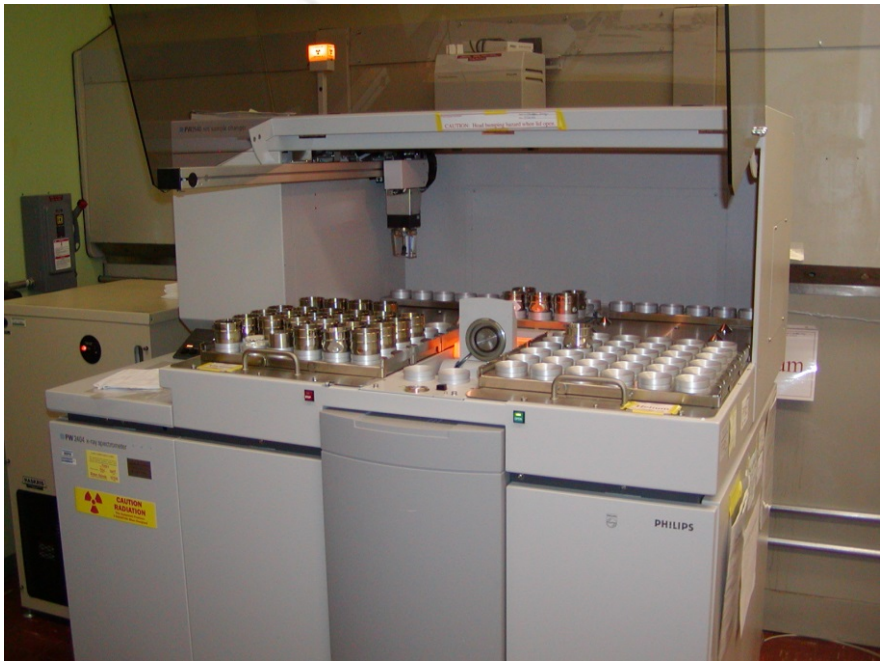
- Description of our XRF capabilities
- Micro-XRF (MXRF) introduction
- MXRF BSAP examples
- Summary

XRF Capabilities

- Wavelength dispersive XRF (WDXRF)
- Polarized energy dispersive XRF (Polarized EDXRF)
- Handheld XRF
- Micro-focused X-ray beam XRF (MXRF)

Wavelength Dispersive XRF (WDXRF)

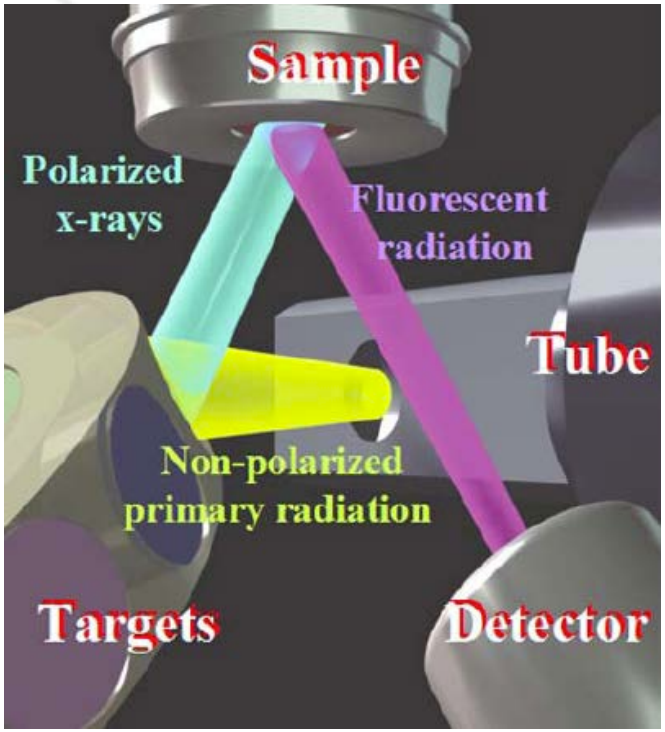
- High power 4000 Watt Rh x-ray tube
- Elemental Range: Boron & Fluorine - Americium
- Advantages
  - Automated: 150 samples can be batched
  - Best sensitivity and reproducibility
  - Best for low Z elements
  - Best for high rad samples – No detector saturation
- Limitations
  - Sequential spectrometer
    - Scan one element at a time
    - Slow for analysis of large set of elements
  - High power X-rays can damage fragile samples
  - Most expensive XRF instrument



Polarized Energy Dispersive XRF (Polarized EDXRF)

- 100 kV X-ray tube
  - Excellent sensitivity for medium & high Z elements
- Elemental range: Sodium to Curium
- 15 secondary target choices
  - Choose target to optimize elemental detection limits (LLDs)
  - Reduced spectral background – Even better LLDs
  - Minimal sample prep – Ratio to secondary target X-ray scatter instead of adding internal standard
- No sample damage
  - Secondary target X-rays do not heat the sample
  - Long count times possible – Ultimate LLDs

- Very high rad samples can saturate the detector
- EDXRF energy resolution not as high as WDXRF
- EDXRF low Z elemental sensitivity worse than WDXRF

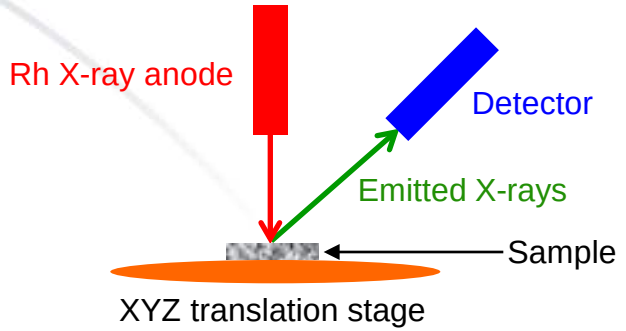


Handheld XRF

- Portable - Take instrument to the sample
- Hold to surface and pull trigger
- Elemental range: Sodium to Americium
- Metal alloy identification
  - Glove box steel grade ID for QA documentation
  - Sample containers alloy ID
- Qualitative analysis of any large object
- Pu and TRU quantification possible with method development

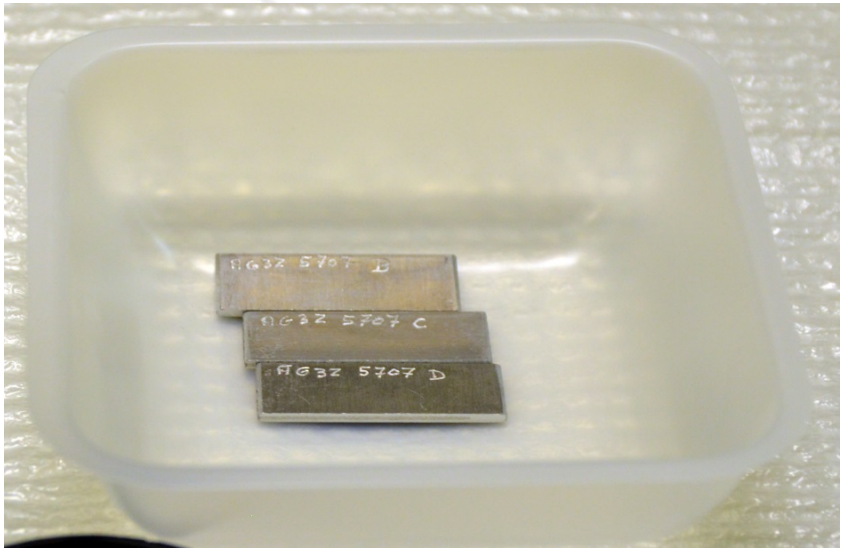
Micro-XRF (MXRF)

- EDAX Orbis instrument
- 30 μm diameter irradiated spot
- 1 & 2 mm spot sizes – Larger samples
- Move sample on stage to image surface elemental distributions
- Elemental range: Sodium to curium
- Image up to 10 cm x 10 cm areas
- X-ray source filters
  - Improves elemental detection limits

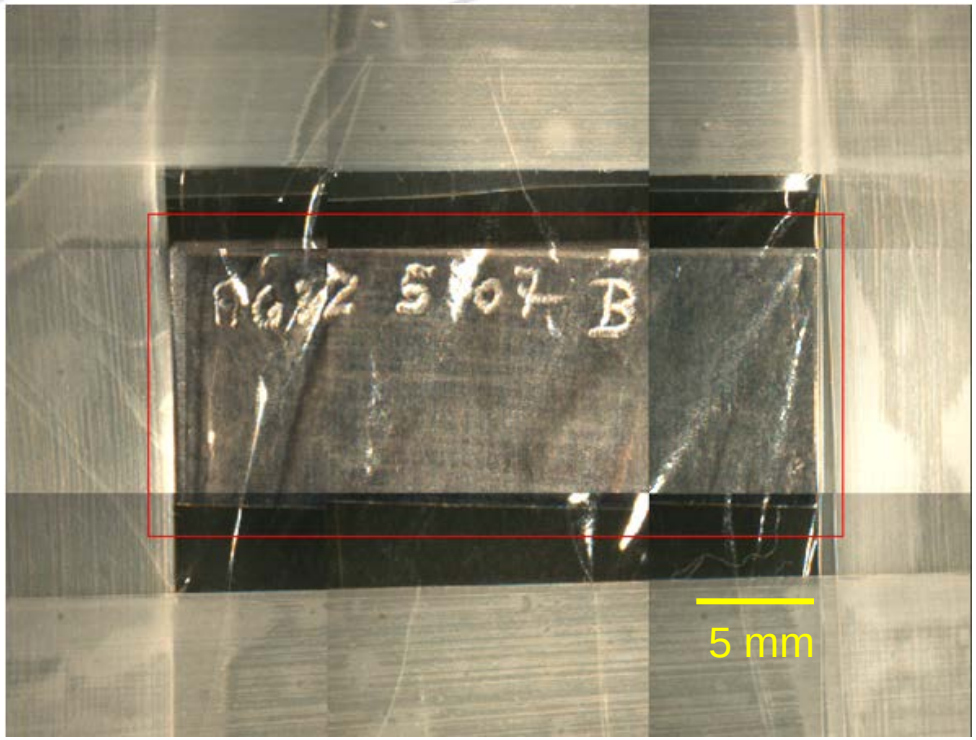


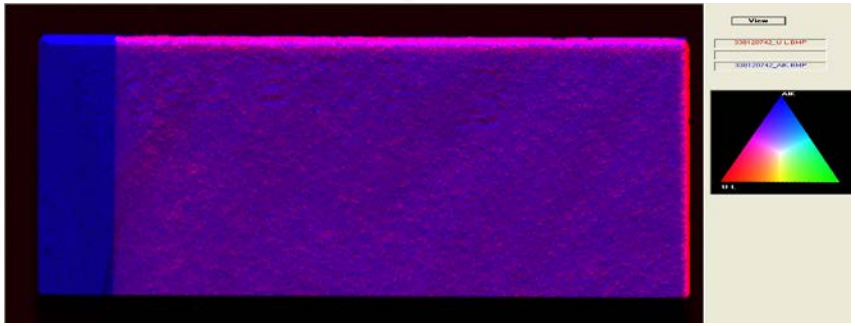
MXRF – HEU / Aluminum Fuel Plates

- HEU/aluminum alloy submitted for comprehensive chemical analysis
- Originally understood the material to be fairly homogeneous
- NDA characterization by MXRF
  - Plate compositions NOT uniform
  - MXRF used to guide where to extract material for further characterization
  - 3D elemental structure - HEU fuel plates with Al cladding; NOT homogenous



MXRF – HEU / Aluminum Fuel Plates





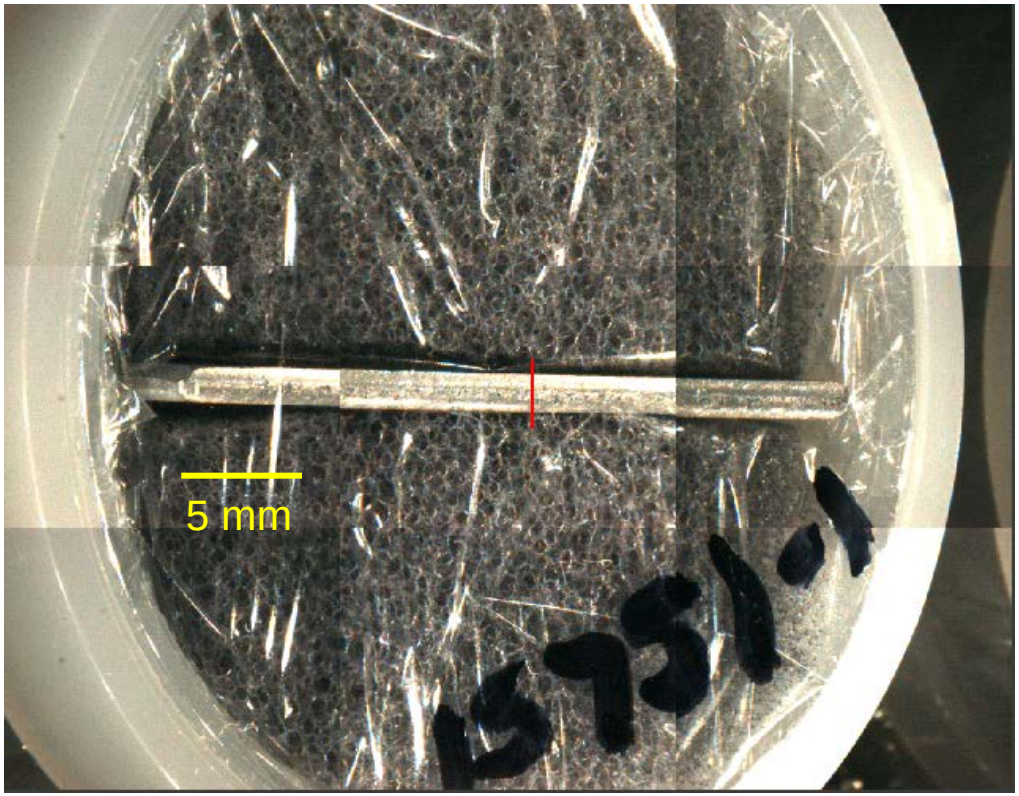
MXRF U & Al image overlay

Blue = Aluminum
Red = Uranium L
Pink = Al & U overlap

- No U on left edge – First evidence of aluminum cladding surrounding U layer
- Next mounted on cut edge to analyze material inside the plate

MXRF – HEU / Aluminum Fuel Plates

HEU/Al plate mounted with cut edge facing up
(Edge cut with sheet metal shear prior to receipt of sample)



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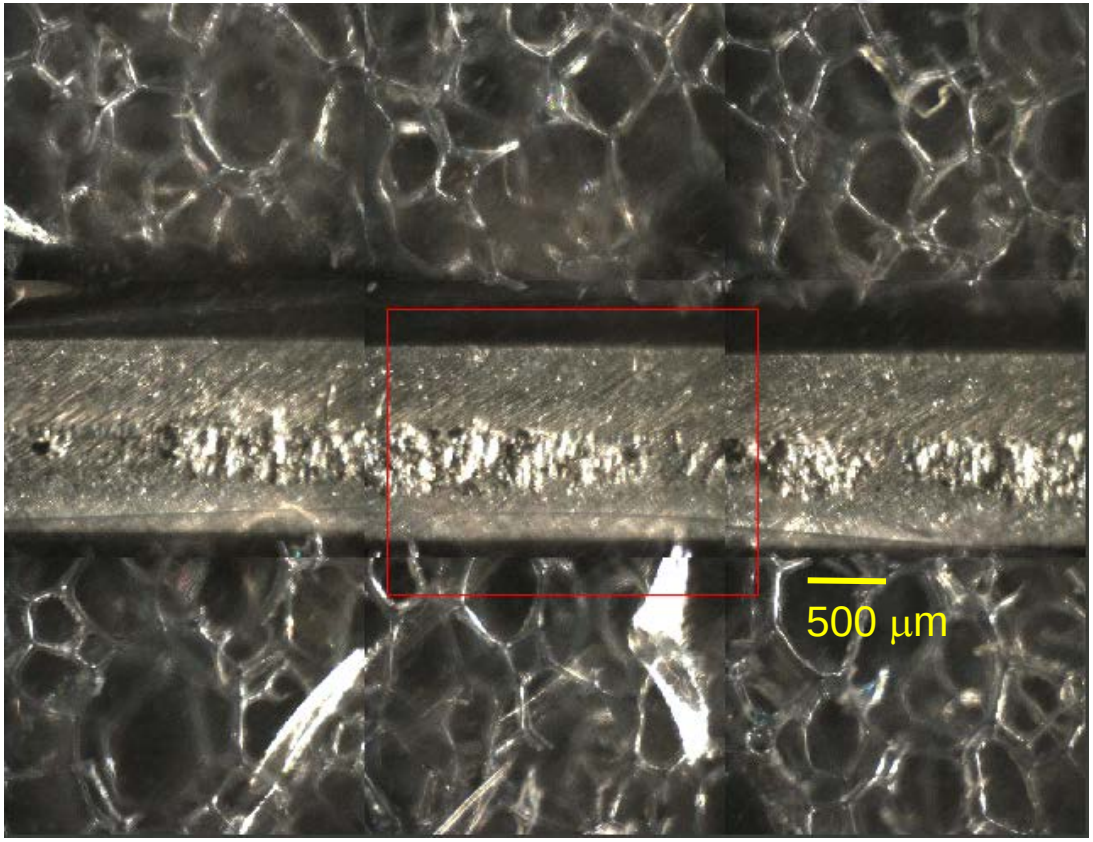
MXRF – HEU / Aluminum Fuel Plates

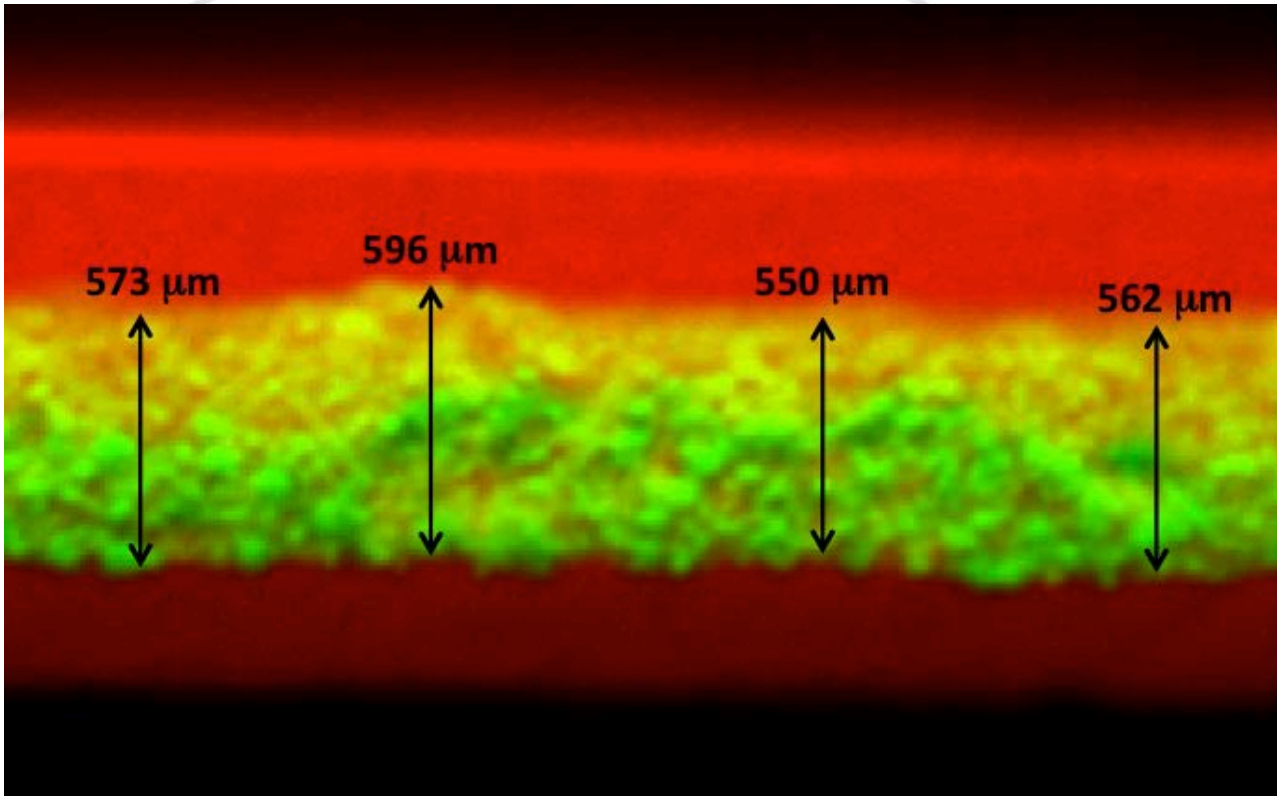


Used MXRF to image red boxed area of plate edge

MXRF – HEU / Aluminum Fuel Plates

Aluminum

Uranium



- Direct confirmation of HEU layer between Al cladding
- Measured U layer thickness
- HEU layer spectral analysis → U & Al alloy

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MXRF – HEU / Aluminum Fuel Plates

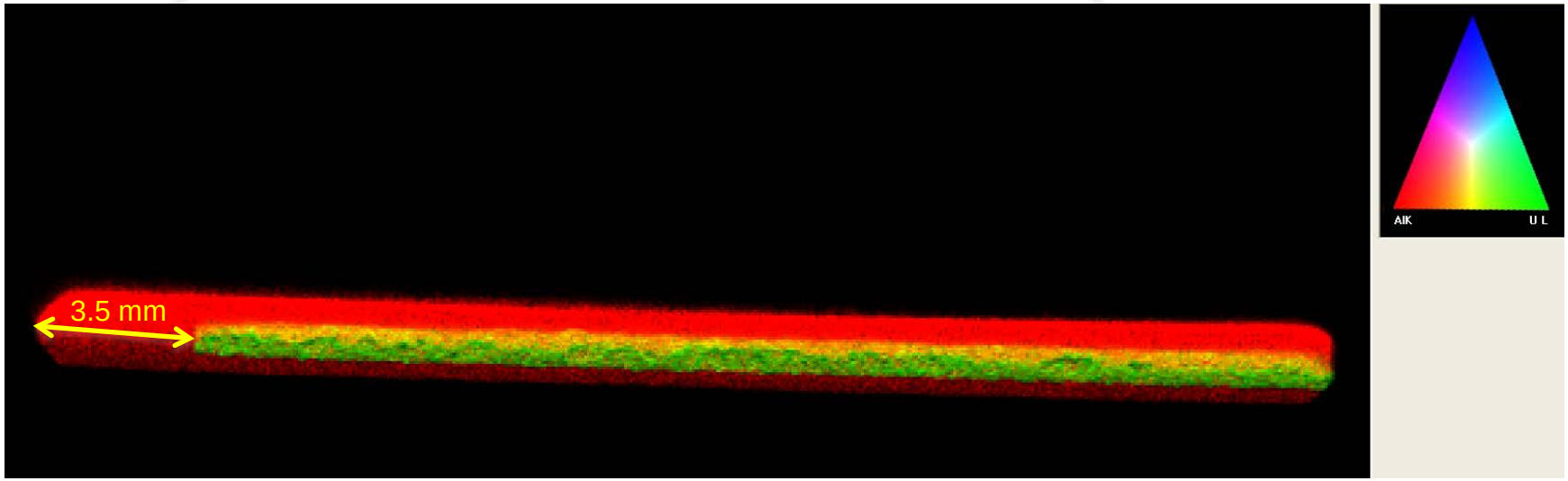


Image of entire cut edge - Aluminum and uranium overlay

Red = Aluminum
Green = Uranium

MXRF – HEU / Aluminum Fuel Plates

- **Standard-less quantification**
  - Approximate concentrations only (high fidelity quant requires standards)
  - Analyzed areas of HEU & Al layers exposed on cut edge using micro-focused X-ray beam
- HEU layer only ~9% Uranium; majority aluminum
- Aluminum layer almost all Al; a few impurities present

Uranium-containing Layer

Element	Approx Wt%
Aluminum	90.7
Uranium	9.1
Miscell impurities	0.2

Aluminum Layer

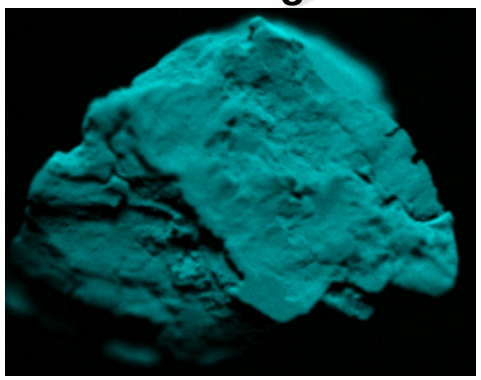
Element	Approx Wt%
Aluminum	98.7
Calcium	0.6
Iron	0.5
Miscell impurities	0.2

MXRF - Electrorefined (ER) Plutonium Metal

- ER Pu metal cuts always extracted from parent for chemical analyses (eg. trace elements by ICP)
- If parent not homogenous, will affect reported chemistry results
- MXRF was examined as an NDA method to detect any parent metal elemental heterogeneity prior to DA

MXRF - ER Plutonium Metal

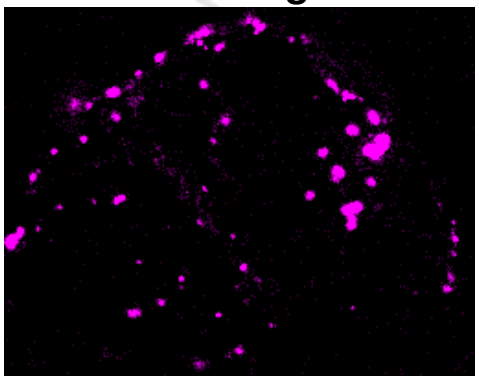
Pu image



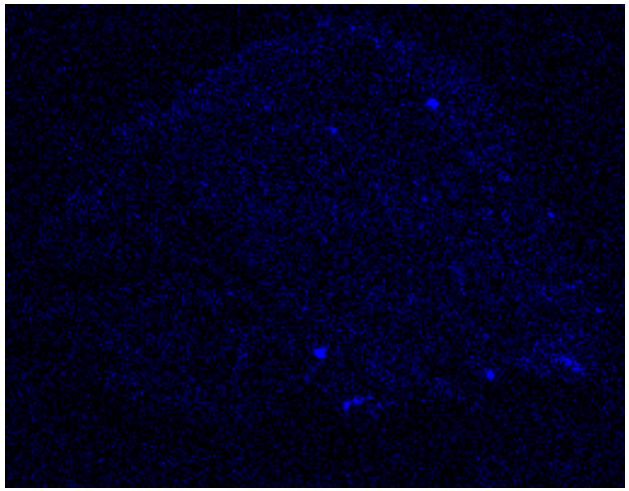
~4.2 mm

350 mg Pu metal chunk

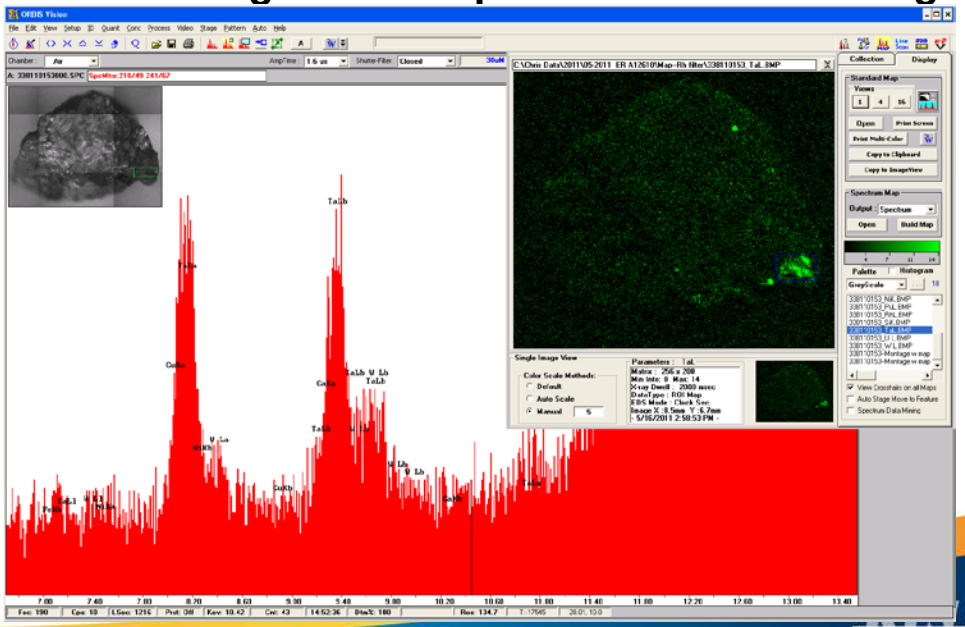
Iron image



Tungsten image



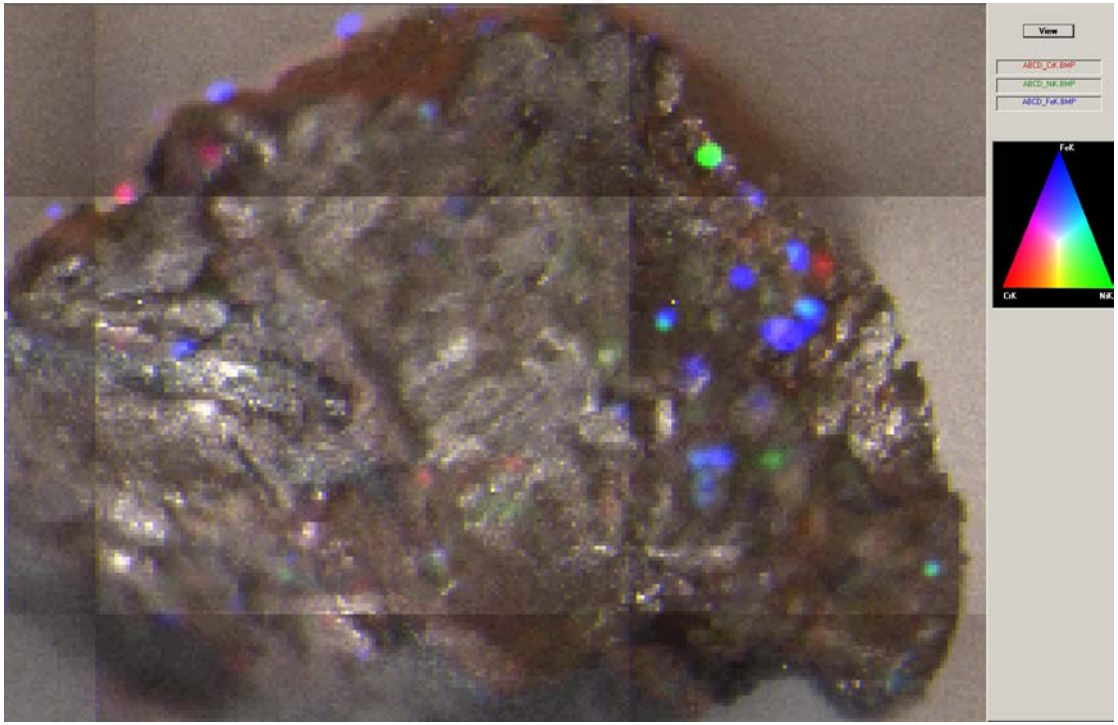
Tantalum image & XRF spectrum from lower right



MXRF - ER Plutonium Metal

- Fe, Ni, Cr spots detected
  - Pu surface oxide removed with steel wire brush
  - A few spots have steel signatures (purple & cyan)
  - Some steel residue from wire brush may be present
  - But other spots are due to a single element (eg. nickel green spot)
  - Some spots indicate sample heterogeneity (ie. pure blue, green, or red)
- If heterogeneity detected by MXRF, avoid T&E repeating ICP prep and analysis

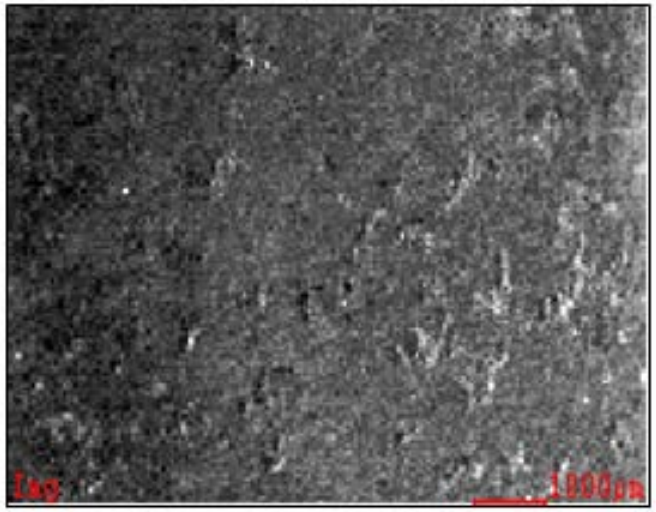
Overlay of Pu metal image with impurity spots

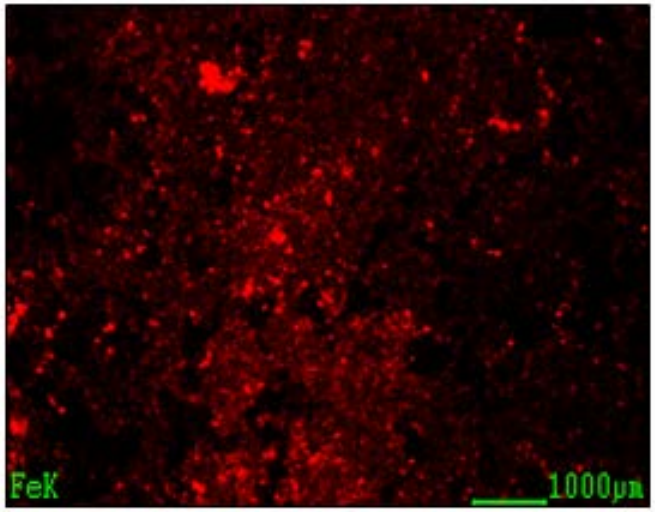


Fe Ni Cr

Cast Pu Metal
MXRF of ~9 mm x ~6 mm area

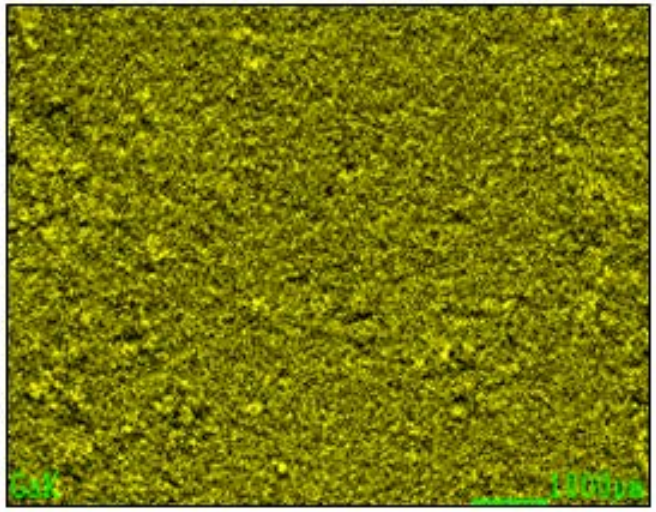
Visible Image





Fe Relative Intensity

Ga Relative Intensity



Pu Relative Intensity

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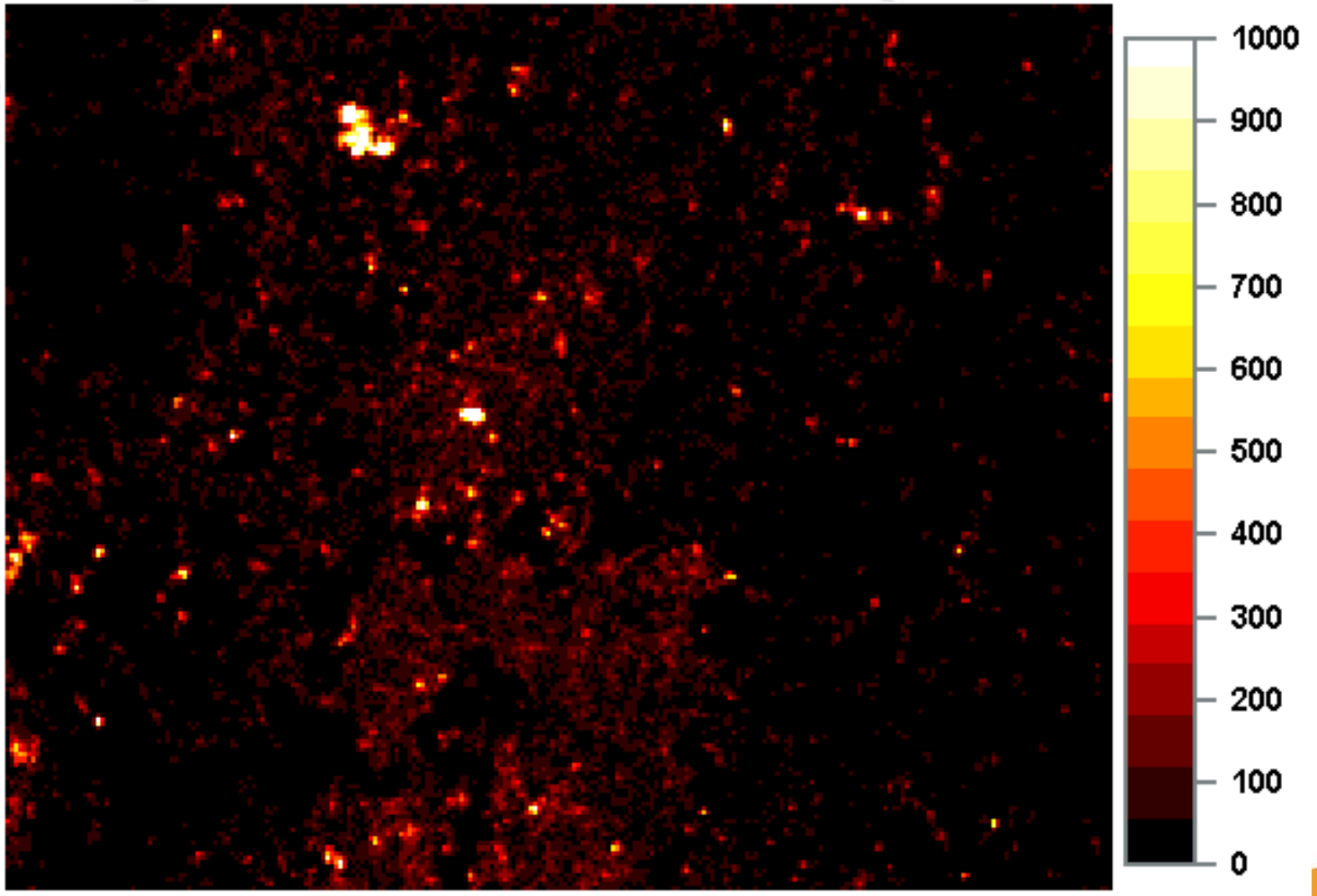
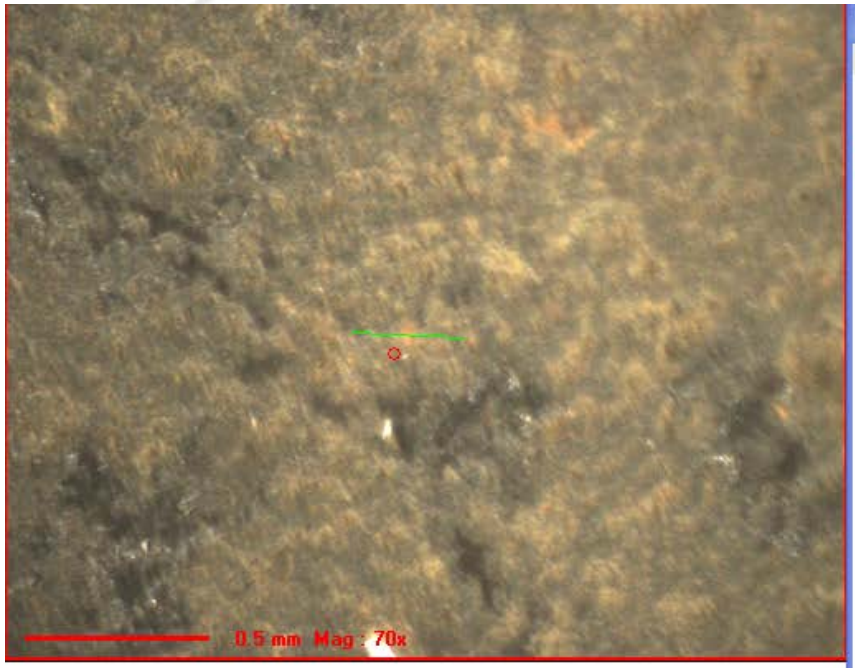
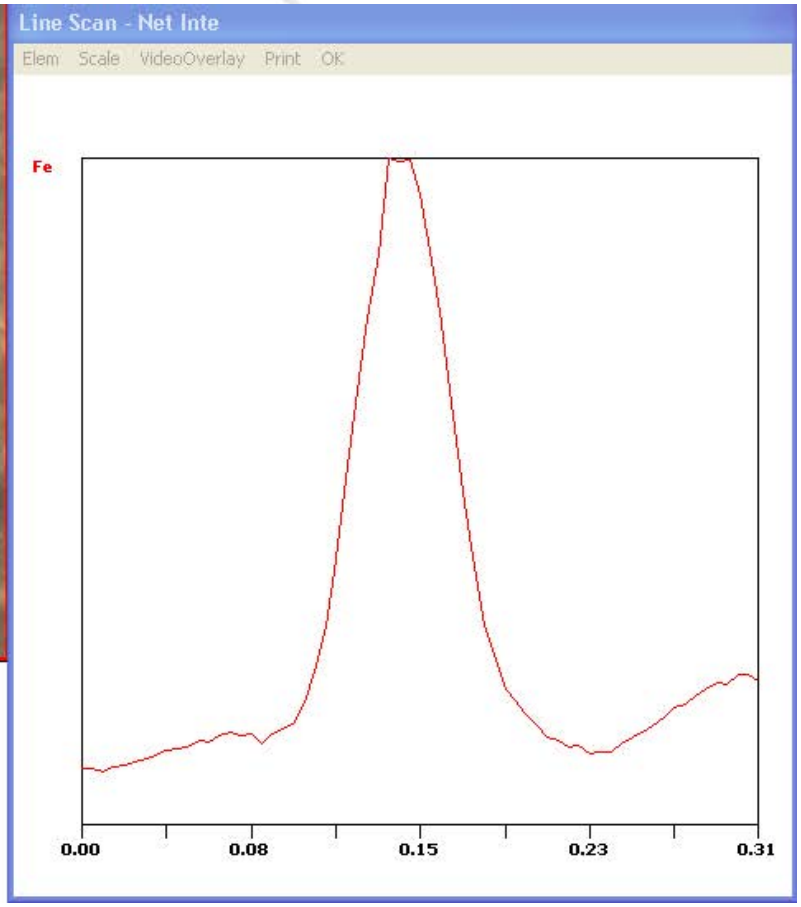
Cast Pu Metal – IRON MXRF MAP



Cast Pu Metal
Smaller Area & Iron Line Scan



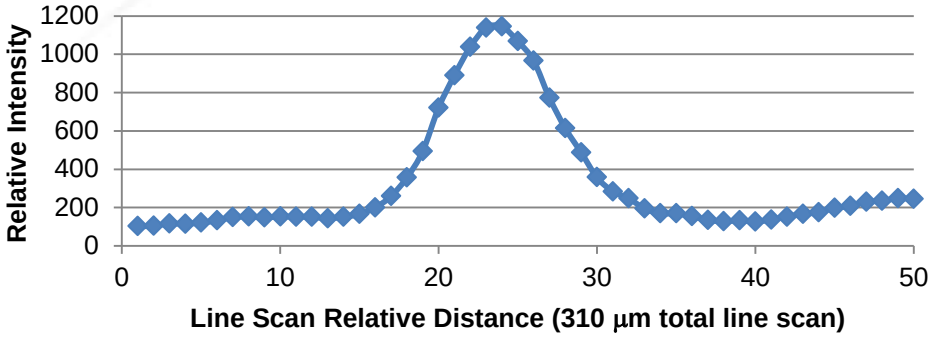
310 μm Line scan in green



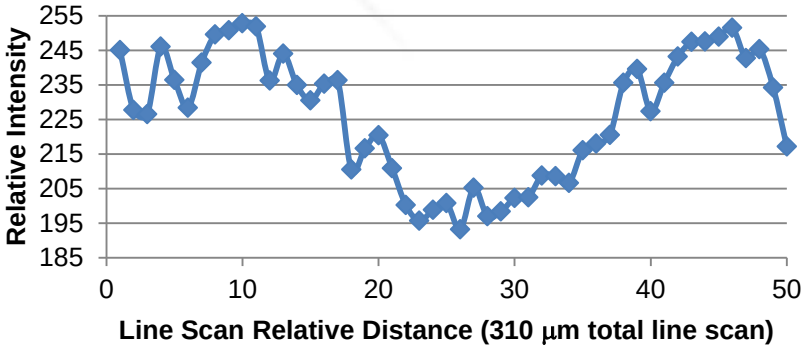
Distance (mm)

Cast Pu Metal MXRF Line Scan Relative Intensities

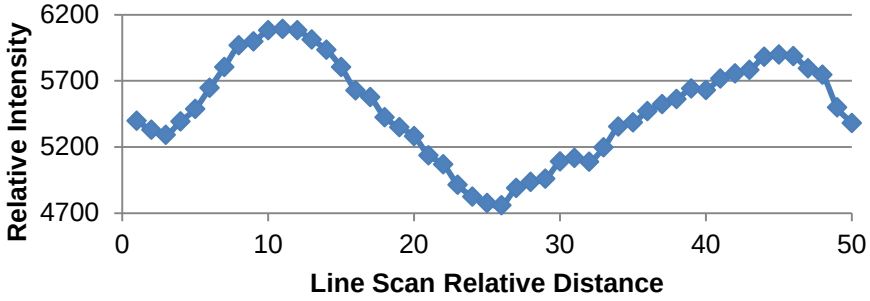
IRON - Relative intensity across line scan



GALLIUM - Relative intensity across line scan



PLUTONIUM - Relative intensity across line scan



Summary

- MXRF complements other imaging techniques such as SEM-EDS
- MXRF method of choice for:
  - Large area analyses (up to 100 cm^2 areas)
  - True NDA – No damage from X-ray beam
  - Medium and high atomic number elemental analyses
  - Non-conducting materials (eg. plastics, HEPA filters, etc.)
  - Samples incompatible with vacuum (eg. liquids, moist samples)
- Presented several MXRF BSAP-related applications
- Other examples of XRF BSAP-related applications
  - Signatures from test debris
  - Gallium and uranium quantification in Pu metals and oxides
  - Alloy identification of NM forensics sample containers
  - Approximate quantification of major & minor elements in Pu oxides